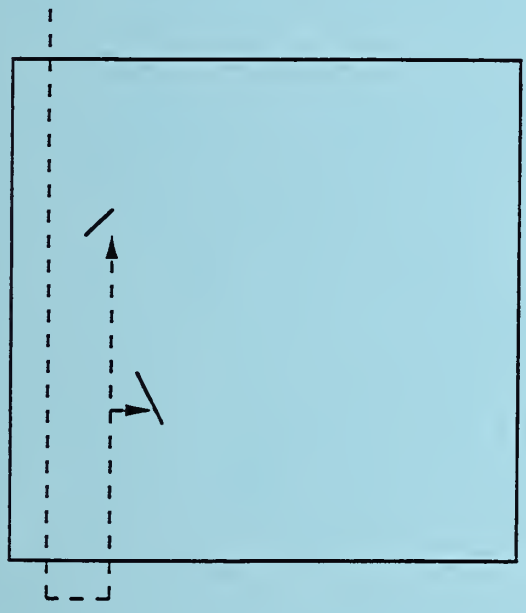
NISTIR 5276

# Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 1.0



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**1. Scope**

1.1 This test method describes a procedure for verified analysis of asbestos by transmission electron microscopy.

1.2 The method is applicable only when sufficient information has been collected during the analyses of a grid square so that individual asbestos structures can be uniquely identified.

1.3 This method is written for the analysis of a grid square by two TEM operators but can be used for more than two operators with slight modifications. Due to the analysis of a grid square by more than one TEM operator, the test method can be applied only when contamination and beam damage of particles are minimized.

1.4 This method refers to the counting rules and associated terminology specified in the AHERA method[1] for asbestos analysis. With slight modifications, this method can be used with any set of counting rules applied by all of the analysts.

**2. Terminology**

2.1 *Definitions:*

2.1.1 *TEM*--transmission electron microscope

2.1.2 *grid square, grid opening*--an area on a grid used for analysis of asbestos by transmission electron microscopy.

2.1.3 *verified analysis*--a procedure in which a grid opening is independently analyzed for asbestos by two or more TEM operators and in which a comparison and evaluation of the correctness of the analyses are made by a verifying analyst. Detailed information -- including absolute or relative location, a sketch, orientation, size (length, width), morphology, analytical information and structure identification -- is recorded for each observed asbestos structure.

2.1.3.1 *Discussion*--Verified analyses can be used to determine the accuracy of operators and to determine the nature of problems that the analyst may have in performing accurate analyses. Verified counts can be used to train new analysts and to monitor the consistency of analysts over time.

2.2 *Description of Terms Specific to This Standard*:

2.2.1 *counting rules*--rules used to determine the number of structures present in an asbestos containing particle.

2.2.2 *AHERA method*--procedure for analysis of asbestos by transmission electron microscopy developed by the Environmental Protection Agency (see footnote 1) with subsequent modifications by NIST.

2.2.3 *TEM operator, TEM analyst*--person that analyzes a grid square by transmission electron microscopy to determine the presence of asbestos.

2.2.4 *verifying analyst*--person that compares the analyses of a grid square by two or more TEM operators. The reported asbestos is compared on a structure-by-structure basis by the verifying analyst. Structures that are not matched are relocated and reanalyzed by the verifying analyst. The verifying analyst is preferably not one of the TEM operators. If this cannot be avoided, the job of verifying analyst should be rotated between the TEM operators.

2.2.5 *count form*--form on which the analysis of a grid square is recorded. The information recorded for a verified analysis should include at least a sketch of the structure and information related to the absolute or relative location, size, identification and analytical data for the reported asbestos structures.

2.2.6 *report form*--form on which the evaluation of verified analyses is summarized. The form should be identical to or include all information given in Figure X1 of the Appendix.

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[1]Code Fed. Reg. **1987**, *52* (No. 210), 41826-41905.

2.2.7 *countable asbestos structure*--a structure containing asbestos that is counted as asbestos under the rules given by the AHERA method.

2.2.8 *SR (structures reported)*--the number of structures reported as countable asbestos by one TEM analyst.

2.2.9 *TNS (total number of structures)*--the number of countable asbestos structures determined to be on a grid opening by verified analysis of the grid opening.

2.2.9.1 *Discussion*--The value for the total number of structures is not necessarily the actual number on the grid square because both TEM analysts and the verifying analyst may have missed one or more structures. The probability of a missed structure, however, decreases with an increased number of analyses.

2.2.10 *TP (true positive)*--countable asbestos structure that is: 1) reported by both TEM operators or 2) reported by one operator and confirmed by the verifying analyst, or 3) not reported by the two TEM operators but is found by the verifying analyst (as discussed in the next term).

2.2.11 *TPV (true positive found by verifying analyst)*--countable asbestos structure not found by the two TEM operators but found by the verifying analyst.

2.2.12 *FN (false negative)*--countable asbestos structure that has not been reported as countable by a TEM analyst. False negatives can be divided into two categories-type A and type B as discussed in the next two terms.

2.2.13 *FNA (false negative-type A)*--false negative that was recorded on a TEM analyst's count form but not reported as countable asbestos. Some reasons for this type of false negative include: 1) structure misidentified as nonasbestos, 2) confusion with the counting rules, 3) incorrect length determination.

2.2.14 *FNB (false negative-type B)*--false negative that was not recorded on a TEM analyst's count form. A reason for this type of false negative is that a structure was missed by an analyst.

2.2.15 *FP (false positive)*--reported structure that is incorrectly identified as countable asbestos. Some reasons for false positives include: 1) structures counted more than one time, 2) structures misidentified as asbestos, 3) confusion with the counting rules, 4) incorrect length determination.

2.2.16 *NL (not located structure)*--structures present on one TEM analyst's count form that cannot be located by the verifying analyst.

2.2.16.1 *Discussion*--The value for NL should be 0 for most verified analyses, especially if the grid has not been removed from the TEM between the two analysts' counts. If, however, a grid has been removed from an instrument, there is a small possibility of fiber loss.

2.2.17 *AMB (ambiguous structure)*--a structure that 1) is identified as countable by one TEM operator and as noncountable by the second TEM operator and 2) is found by the verifying analyst but cannot be unambiguously identified as a countable structure due to beam damage or contamination.

**3. Significance and Use**

3.1 The analysis of asbestos by transmission electron microscopy is important for determination of the cleanliness of air or water and for research purposes. The test method provides a procedure for determining the quality of the analyses.

3.2 The test method can be used as part of a quality assurance program for asbestos analyses and as a training procedure for new analysts. The values for TP/TNS and FP/TNS can be plotted *vs* time on control charts to show improvements or degradations in the quality of the analyses. Experienced analysts should attain TP/TNS values $\geq 0.85$ and FP/TNS values $\leq 0.05$.

3.3 The average of values obtained for TP/TNS and FP/TNS can be used in determining the analytical error for routine asbestos analyses.

**4. Procedure**

NOTE 1-- This test method involves two TEM operators and a verifying analyst. The steps discussed in items 4.1 and 4.2 are to be followed by the person coordinating the analyses by the TEM operators. This person can be one of the TEM operators, the verifying analyst or an independent person (e.g., a quality assurance officer). The steps discussed starting with item 4.3 are to be followed by the verifying analyst.

4.1 Obtain analyses of a grid square for asbestos by two TEM operators. Conduct the analyses independently so that the second operator has no knowledge of the results obtained by the first operator.

4.1.1 Require that the TEM operators record on the count form information related to the absolute location of the analyzed structures or conduct analyses so that the relative location of the analyzed structures can be compared.

NOTE 2-- The absolute location of the analyzed structures can be recorded by various means including use of a digital voltmeter or computer readable stepping motors to record the position of a structure. To preserve information about the relative location of the reported structures, the analyses must be conducted so that both analysts: 1) orient the grid in the TEM in the same fashion, 2) start the analysis from the same corner of the grid square, 3) initially scan in the same direction, and 4) scan the grid square in parallel traverses.

4.1.2 Require that the TEM operators record on the count form a sketch of the structure, size, morphology, and analytical data.

4.2 Submit the analyses of the two TEM operators to a verifying analyst.

NOTE 3-- The remainder of this section describes procedures to be followed by the verifying analyst.

4.3 Count the number of asbestos structures obtained by each analyst and enter the value as SR (structures reported) on the report form. Care should be taken to determine the number of asbestos structures reported for any given sketch on the analysis form (for example, one sketch may correspond to 1, 2 or 3 structures).

4.4 Compare the two analyses on a structure-by-structure basis. If a match of asbestos structures is observed, place the same number next to the sketches of the structures or in a column specifically designated for verified counts (i.e., put 1's in the appropriate location on both count forms for the structures that correspond to the first recognized match, 2's in the appropriate location on both count forms for the structures corresponding to the second recognized match, etc.). All matched asbestos structures are considered true positives.

NOTE 4-- To qualify as a match, the structures should be comparable in the following characteristics: 1) absolute or relative location, 2) appearance in the sketch, 3) orientation, 4) size (length, width), 5) morphology (shape, hollow tube), 6) analytical information (chemistry and/or diffraction data), and 7) structure identification. Care should be taken to check the number of structures reported by the analyst on each line and match each of the reported structures (i.e., one line may contain one, two or three numbers depending on the number of structures).

4.5 For asbestos structures for which no match has been found or for which discrepancies occur, check the grid square in the TEM to investigate the reported structure.

4.5.1 If the reported structure is not found, place an NL in the appropriate place on the count form (next to the sketch or in a column specifically designated for verified analyses).

4.5.2 If the reported structure is found, attempt to determine its identity and to determine if it is a countable asbestos structure.

4.5.2.1 If the reported structure cannot be unambiguously determined to be a countable structure, place an AMB in the appropriate location on the count form.

4.5.2.2 If the reported structure is incorrectly identified as countable asbestos, place an FP(number) in the appropriate location on the count form. A unique number is given to the FP label so that it can be specifically referred to in the report form.

4.5.2.3 If the reported structure is correctly identified as countable asbestos, place a TP(number) in the appropriate location on the count form. Determine if the other TEM operator reported the structure as noncountable or did not report the structure on their count form.

4.5.2.3.1 If the other TEM operator reported the structure as noncountable, write FNA(number) on their count form in the appropriate location on the TEM operator's count form (next to the sketch or in a column specifically designated for verified analyses). The number should correspond to that given to the TP on the first analyst's count form.

4.5.2.3.2 If the other TEM operator did not report the structure on his/her count form, write FNB(number) on their count form in the approximate location where the structure should have been found. The number should correspond to that given to the TP on the first analyst's count form.

4.5.2.4 For most cases, the identification of true positives and false positives can be done on a structure-by-structure basis. In some instances, however, this cannot be done. For example, a TEM operator may incorrectly identify a cluster as three or more structures rather than as one structure. The first structure identified by this operator should be matched with the cluster identified by the other operator. The remaining structures identified as countable should be designated as false positives. Similarly, a TEM operator may incorrectly count each of the fibers protruding from a matrix as a countable structure rather than counting the entire arrangement as one structure. The first structure identified by this operator should be matched with the matrix identified by the other operator and the remaining structures in this arrangement should be identified as false positives.

4.6 Countable asbestos structures reported by neither TEM operator but found by the verifying analyst in the course of examining a grid square should be recorded on a separate count form and labelled TPV(number). The TEM operators should be assigned an FNA(number) or FNB(number) as described in items 4.5.2.3.1 and 4.5.2.3.2.

4.7 Determine the total number of true positives (TP) obtained for each operator on the grid square and enter the value onto the report form (under TP). The total number of true positives for any analyst will correspond to the number of matched structures plus the number of structures with TP written next to them.

4.8 Determine and record on the report form the number of true positives found by the verifying analyst (TPV).

4.9 Determine and record on the report form the total number of structures (TNS) on the grid square and the number of matched structures in the two analyses.

4.10 Determine and record on the report form for each operator the following: 1) the number of false positives (FP), 2) the number of false negatives (FN), 3) the number of false negatives of type A and type B (FNA, FNB), 4) the number of structures that were not located (NL) and 5) the number of ambiguous structures (AMB).

4.11 Determine and record the values for TP/TNS, FP/TNS to two decimal places.

4.12 List on the report form the suspected reasons for the false positives obtained by each analyst. Some examples would be as follows: incorrect length measurement, structures counted

twice, problem with interpretation of the counting rules, misidentification of a structure.

4.13 List on the report form the suspected reasons for false negatives (FNA and FNB). Some examples would be: incorrect length measurement, problem with interpretation of the counting rules, misidentification of a structure, possible loss of sense of direction, and insufficient overlap of traverses.

4.14 Append any other relevant comments to the report form (quality of the preparation, etc.).

4.15 Check the numbers on the report form using the equations given in the calculation section.

**5. Calculation**

5.1 The values on the report form should be consistent with the following equations:

For both analyses:

$$TNS = (TP\ [analyst\ 1] + TP\ [analyst\ 2]) - \#\ of\ matches \quad (+ TPV)$$

For a given analysis:

$$SR = TP + FP \quad (+ NL + AMB)$$

$$TNS = TP + FN$$

$$1 = TP/TNS + FN/TNS$$

**6. Precision and Bias**

6.1 To determine the precision of the method, independent verified analyses were conducted on a set of 21 grid squares. The mean value for TNS for the data set is 16.2 structures/grid square and the pooled standard deviation of the pairs of verified count determinations is 1.12 structures/grid square. The confidence at approximately the 95% level of a reported verified count value in this data set is 13.9%.

NOTE 5-- The differences in the values obtained for the independent verified analyses described in item 6.1 are for the most part due to differences in interpretation of the counting rules. The structures analyzed in the study were complex and therefore the precision value discussed above likely represents an upper bound to the precision for the method.

6.2 The bias in the method will vary depending upon interpretation by the TEM operators and verifying analyst of the counting rules used in the analysis.

**7. Keywords**

7.1 asbestos; quality assurance; transmission electron microscopy; verified analysis

## APPENDIX

**(Nonmandatory Information)**

## X1. TEST REPORTING FORM

Fig. X1.1 The following format is suggested for use by the verifying analyst to report the comparison of verified counts.

Grid box: _____ Date: _____

Grid slot: _____ Verifying Analyst: _____

Grid square: _____

| | Analysis 1 | Analysis 2 |
|---|---|---|
| TEM Analyst (TEM operator) | | |
| Structures Reported (SR) | | |
| True Positives (TP) | | |
| TPV | | |
| # of matches of TP | | |
| Total # Structures (TNS) | | |
| False Positives (FP) | | |
| False Negatives (FN) | | |
| FNA | | |
| FNB | | |
| Not Located (NL) | | |
| Ambiguous (AMB) | | |
| TP/TNS | | |
| FP/TNS | | |

Testing Report Form (continued)

1) List details of suspected reasons for false positives. For each analyst describe reasons for FP1, FP2, FP3, etc. Note - it may not be possible to determine the reason for false positives for some structures.

2) List details of suspected reasons for false negatives (type A and type B). For each analyst describe reasons for FNA-1, FNA-2, etc.; FNB-1, FNB-2, etc. Note - it may not be possible to determine the reasons for false negatives for some structures.